

Biodiesel Quality

**from feedstock to formulation
(from Avocado to Liposuction)**

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Need for standards

- Why Standards?
- What is the purpose of Standards?
- Which Standards?
- Who develops/maintains the Standards?
- Who uses the Standards?
- Who accredits / tests user performance?

Standards and specifications

- For engine performance
- For biodiesel quality
- Measurement of input/output quality
 - For feedstock quality
 - For methyl ester quality

Who develops the standards?

International Organizations in the US

- EMA
- ASTM International
- AOCS

Who develops the standards?

Other development & harmonization activities

- Several presentations over the past 1.5 days for many other regional activities
- ISO/TC 34/SC 11 and TC 28 proposed Joint Working Group (ballot closes soon)
- ISO TMB Resolution for ANSI and ABNT to include a new SC to ISO/TC 28 for Biofuels

AOCS Mission

- a forum for the exchange of ideas, information and experience
- professional interest in the science and technology of fats oils and related substances
- promote excellence and provide high standards of quality

AOCS Structure

Publications

Membership

**Meetings
and
Exhibits**

**Technical
Services**

AOCS Technical Services Expertise

- World authority in the development and implementation of methods of analysis for fats, oils and oilseeds
- Publisher of *Official Methods and Recommended Practices of the AOCS*
- Provider of the AOCS Laboratory Proficiency Program (LPP)
- US Administrator for ISO TC 34 (Food Products)

Feedstock Assessment

- AOCS methods are prescribed for quality determinations in the tallow and grease industry specifications
- equally important in feedstock assessment considering the wide range of fat-containing products which may be used as source material

AOCS Methods for Tallow and Grease Quality Parameters

Color

a. FAC

Cc 13a-43

b. R & B Color

Cc 8d-55

Fatty acid profile

Ce 1h-05

Free fatty acids

Ca 5a-40

Iodine value (IV)

Cd 1d-92

Lead content

Ca 18c-91

Moisture

Ca 2c-25 / Ca 2b-38

Unsaponifiable matter

Ca 6a-40

Insoluble impurities

Ca 3a-46

Peroxide value (PV)

Cd 8b-90

Polyethylene (PE)

Ca 16-75

Rate of filtration

Saponification value (SV)

Cd 3-25

Titer

Cc 12-59

- Methods in **bold** text are recommended for use in the Tallow and Grease Series of the AOCS Laboratory Proficiency Program (LPP), respectively.

Why is it important to assess the Feedstock Quality?

- Pretreatment can be critical and can increase the cost of production
- Need for complete reaction to FAMEs
 - Glycerin removal
 - Catalyst removal
 - Alcohol removal
 - Free Fatty Acid

AOCS Biodiesel Expert Panel

Aim:

Quality, from feedstock to formulation

AOCS Biodiesel Expert Panel (2)

Priorities

- Improve current analytical methodologies where necessary
- Identify laboratories/invite participation in quality schemes
- Identify rapid and field test applications
- Expert panel to consider official method harmonization

Analytical Priorities

- Guidelines for feedstock quality
 - New Recommended Practice published April 2007
- Free, total glycerol...etc by GC/FID
 - Proposed method for collaborative study
- Glycerol enzymatic
 - Alternative method for glycerin
 - Potential for collaborative study

Analytical Priorities (2)

- Acid value
 - B100, B20, B5, samples circulated and tested by different methods: ASTM 664, 974, CEN 14104 and AOCS Cd 3d-63
 - In progress, study report available 3rd quarter
- Leftover diesel catalysts on biodiesel blend stability and Trace metals known to affect edible oil stability in diesel fuels by ICP
- Vacuum/simulated distillation

Analytical Priorities (3)

- % ester to test for major contaminants
- Precipitates and cold flow
- Stability
- Peroxide values of raw materials
- Flash point
 - AOCS to adopt changes to ASTM D 93 method C

AOCS Recommended Practice

Ck 1-07

Recommended Practices for Assessing Feedstock to Ensure Biodiesel Quality

lists those methods which may be used to assess the quality of oils and fats used in the production of biodiesel.

SCOPE

restricted to triglyceride feedstocks for biodiesel manufacture , including vegetable oils (soybean oil, rapeseed oil, palm oil, etc.), animal fats (tallow, lard, etc.) and triglyceride greases (yellow grease, etc.).

Critical attributes of the triglycerides are considered in light of the requirements of the most common biodiesel process - the alkaline transesterification of the triglyceride feedstock with methanol to produce fatty acid methyl esters.

Ck 1-07 (2)

- Sampling
- Cleanliness
- Purity
- Impurities
- Oxidative Stability
- Fatty Acid Composition

Table 1. Methods to assess feedstock quality.

Test	Method
Sampling	AOCS C 1-47
Insoluble impurities	AOCS Ca 3a-46
Sediment by centrifugation	AOCS Ca 3d-02
Unsaponifiable matter	AOCS Ca 6b-53
Polar compounds in frying fats	AOCS Cd 20-91
Soap in Oil	AOCS Cc 17-95
Polymerized triglycerides by gel-permeation HPLC	AOCS Cd 22-91
Water by modified Karl Fischer method	AOCS Ca 2e-84
Modified moisture and volatiles	AOCS Ca 2f-93

Table 1. Methods to assess feedstock quality (2)

Acid value	AOCS Cd 3d-63
Sulfur - elements in oil by ICP-OES (use general guidance)	AOCS Ca 17-01
Phospholipids in vegetable oils	AOCS Ca 19-86
Phosphorus in oil by ICP-OES	AOCS Ca 20-99
Phosphorus content – colorimetric method;	AOCS Ca 12a-02
Phosphorus	AOCS Ca 12-55
Fat stability, Oil Stability Index (OSI)	AOCS Cd 12b-92
Fat stability, peroxide value	AOCS Cd 8-53 or Cd 8b-90
p-Anisidine value	AOCS Cd 18-90
Polymerized triglycerides by gel-permeation HPLC	AOCS Cd 22-91
Polar compounds in frying fats	AOCS Cd 20-91
Fatty acid composition	AOCS Ce 1-62
Methyl ester preparation	AOCS Ce 2-66

Biodiesel Expert Panel Next Steps

- Implement AOCS Recommended Practice Ck 1-07 for Feedstock Assessment through a new Laboratory Proficiency Program series
- Rapid Methods?
- Other method improvements?

AOCS involvement through ISO

- ISO TC 34 SC11 (fats and oils) resolutions from May 2004 indicate a need for parallel work be initiated at ISO by USA for methods of analysis concerning the quality parameters of biodiesel feedstocks
- AOCS initiated discussions with NBB and the ASTM's Biodiesel Task Force to formulate the US position

ISO (2)

- NWIP was submitted and balloted with approval through SC11
- ISO TMB suggested a JWG with TC28 to mirror the activities at CEN (JWG TC19/307)
- AOCS initiated discussions with Secretary of TC28

ISO (3)

- ISO TMB resolution May 2007 indicates the need for a new Biofuels SC for ISO TC 28
- The SC will be organized by ANSI and ABNT
- Formal scope is under development

Thank you!

For more information or to become involved please
contact:

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**International Congress on Biodiesel:
The Science and The Technologies**

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